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(54) **Method of refining a melt of aluminium scrap melt and aluminium alloy obtained from the refined melt**

(57) A method of refining a melt of aluminium scrap material which comprises metallic aluminium and impurities including iron, in order to obtain a target iron level, comprises

(i) determining the initial amounts, in % by weight, in the melt of Mn, Fe and Si, these amounts being  $[Mn_0]$ ,  $[Fe_0]$  and  $[Si_0]$ ; and

(ii) adding a quantity  $Mn_x$  of Mn to the melt, so as to obtain in the melt, after the refining of the melt, a ratio  $\delta$  given by

$$\delta = [Mn_1] / [Fe_1]$$

wherein  $[Mn_1]$  and  $[Fe_1]$  are the amounts in % by weight of Mn and Fe after the refining of the melt,  $[Fe_1]$  being the desired target level of Fe and  $[Mn_1]$  being given by

$$[Mn_1] = A - B * [Fe_1]$$

wherein

$$1.86 - 0.17 * [Si_0] + 0.004 * [Si_0]^2 < A < 2.21 - 0.17 * [Si_0] + 0.005 * [Si_0]^2$$

and

$$0.42 + 0.50 * \exp(-0.28 * [Si_0]) < B < 0.57 + 0.50 * \exp(-0.28 * [Si_0]).$$

The added quantity  $Mn_x$  of Mn is given by

$$Mn_x = \delta * Fe_0 - Mn_0$$

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wherein  $Fe_0$  and  $Mn_0$  are the initial quantities of Fe and Mn in the melt.

Thereafter the method comprises homogenizing the melt by heating, cooling the melt and maintaining it at a super-eutectic holding temperature so that solid intermetallic compounds form, and separating the solid intermetallic compounds.

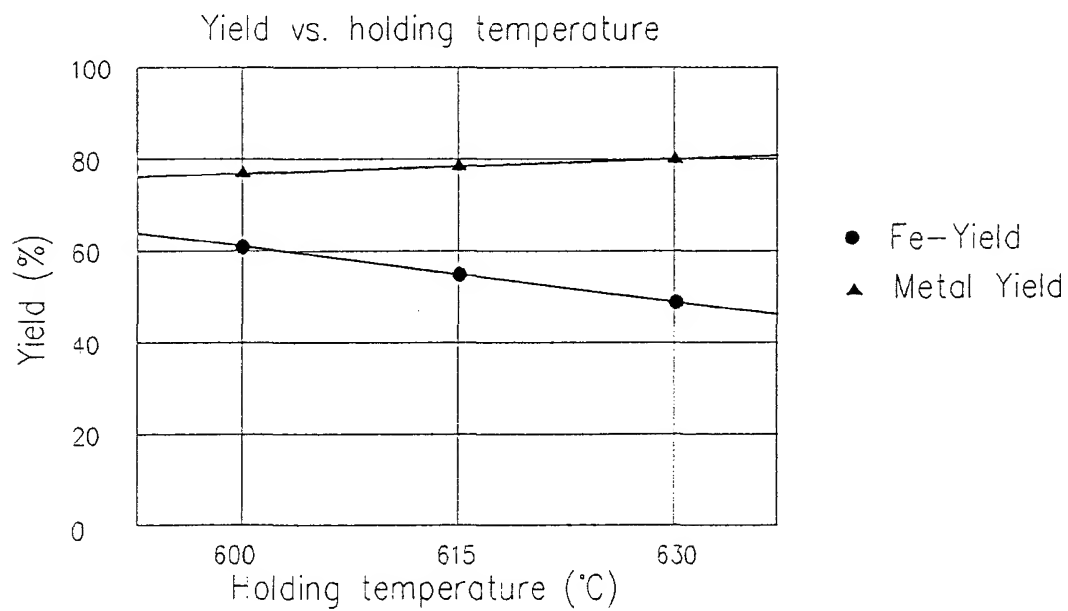


Fig. 10

**Description**FIELD OF THE INVENTION

The invention relates to a method for refining a melt of aluminium scrap material which comprises metallic aluminium and also impurities including iron. This melt is obtained by melting aluminium scrap material. The invention also relates to the aluminium alloy obtained from the refined melt produced by the method.

DESCRIPTION OF THE PRIOR ART

It is known to refine an aluminium melt to remove unwanted metallic impurities by allowing separable solid inter-metallic compounds to form. These can be filtered off. US-A-2464610 describes such a process, in which the Fe content of an aluminium-silicon alloy is reduced to less than 0.5% by adding at least one of Mn, Co and Cr in amounts equal to about 0.5 to 2 times the total weight of Fe present, and slowly cooling the alloy to separate the solidus containing a major part of the Fe at a temperature above the eutectic. Another general disclosure of this type is FR-A-976205.

In SU-1108122 (as abstracted by WPI/Derwent), Mn is added to produce low Fe alloy. The amount of Si in the melt is thereby much reduced.

A study of the formation of these intermetallic compounds in Al-Si alloys is to be found in Z. Metallkunde 86 (1995) 7, pages 457-464, which describes the crystallization of coarse sludge particles at high Mn levels.

WPI/Derwent abstracts of JP-A-7-70666 and JP-A-6-234930 describe separation of Al-Fe-Mn-Si intermetallic compounds to reduce Fe levels, and WPI/Derwent abstracts of JP-A-7-54070, JP-A-6-299265 and JP-A-7-54063 show apparatus for carrying out this aluminium refining.

A drawback of refining methods known in practice is that the yield from refining, as expressed in the attainable degree of removal of, in particular, iron, is low. Another drawback is that Mn is overdosed in order to obtain with reasonable certainty a melt which is sufficiently refined in terms of iron.

SUMMARY OF THE INVENTION

The object of the invention is to provide a method of refining a melt of aluminium scrap material, bearing in mind the varying iron contents which may exist in such melts, which achieves with adequate precision a desired low level of Fe.

A further object of the invention is to provide a method of refining a melt of aluminium scrap material which can avoid use of an excessive amount of Mn.

According to the invention there is provided a method of refining a melt of aluminium scrap material which comprises metallic aluminium and impurities including iron, comprising the steps of

(i) determining the initial amounts, in % by weight, in the composition of the melt of the elements Mn, Fe and Si, these amounts being identified below as  $[Mn_0]$ ,  $[Fe_0]$  and  $[Si_0]$ ;

(ii) adding a quantity  $Mn_x$  of Mn to the melt, so as to obtain in the melt, after refining of the melt by steps (iii), (iv) and (v) below, a ratio  $\delta$  given by

$$\delta = [Mn_1] / [Fe_1]$$

wherein  $[Mn_1]$  and  $[Fe_1]$  are the amounts in % by weight of Mn and Fe in the melt after the refining of the melt,  $[Fe_1]$  being a desired target level of Fe and  $[Mn_1]$  being given by

$$[Mn_1] = A - B * [Fe_1]$$

wherein

$$1.86 - 0.17 * [Si_0] + 0.004 * [Si_0]^2 < A < 2.21 - 0.17 * [Si_0] + 0.005 * [Si_0]^2$$

and

$$0.42 + 0.50 * \exp (- 0.28 * [Si_0]) < B < 0.57 + 0.50 * \exp (- 0.28 * [Si_0]),$$

the added quantity  $Mn_x$  of Mn being given by

$$Mn_x = \delta * Fe_0 - Mn_0$$

wherein  $Fe_0$  and  $Mn_0$  are the initial quantities of Fe and Mn in the melt of aluminium scrap material;

(iii) homogenizing the melt by heating after step (ii);

(iv) cooling the melt after step (iii) and maintaining the melt at a super-eutectic holding temperature T for a holding time t so that solid intermetallic compounds form;

(v) separating the solid intermetallic compounds from the melt, to obtain the refined melt.

In these mathematical expressions, \* denotes multiplication.

The method of the invention is based on selection of the desired end level of Fe,  $[Fe_1]$ , and addition of the appropriate quantity of Mn,  $Mn_x$ , to achieve this end level on the basis of the initial Si content,  $[Si_0]$ , also. It will be apparent that the quantities  $Fe_0$ ,  $Mn_0$  and  $Mn_x$  are expressed in suitable weight units, e.g. kg.

It has been found that the method in accordance with the invention can sufficiently reduce the content of impurities, in particular iron, without use of an excess of, for example, Mn. This permits a re-usable alloy to be made from aluminium thus refined without any further special measures. Apart from increasing the possible applications for manufacturing re-usable alloys, the absence of the excessive use of additives, in particular Mn, reduces costs.

An important advantage of no excess of Mn is also that in principle no more intermetallic compound than necessary is formed, so that the separation stage is less heavily loaded and this results directly in a higher metal yield.

By working in accordance with the invention it is now possible to arrive precisely at a desired refined composition, without any excess of additives needing to be added. It will be clear that if the quantity  $(\delta * Fe_0 - Mn_0)$  given above is negative, then no Mn needs to be added; there is then more Fe removed than is required for attaining the desired  $[Fe_1]$ .

Preferably B is given by

$$0.45 + 0.50 * \exp (- 0.28 * [Si_0]) < B < 0.50 + 0.50 * \exp (- 0.28 * [Si_0]).$$

Preferably, if the melt is an Al-Si12-Fe-Mn system, A lies between 0.76 and 0.80 and B is approximately 0.49. If the melt is an Al-Fe-Mn system preferably A lies between 2.00 and 2.04 and B is approximately 0.96, and if it is an Al-Si8-Fe-Mn system preferably A lies between 0.97 and 1.01 and B is approximately 0.52.

In the method, preferably the separating takes place in a filter with a filter porosity p less than 30 ppi (pores per square inch). This permits a very good Fe removal yield ( $\eta_{Fe}$ ) to be achieved.

The Mn may be added in a conventional manner, e.g. as an aluminium alloy.

#### DESCRIPTION OF TEST EXAMPLES

The invention will now be explained and illustrated by reference to test examples.

These test examples were designed to simulate, under controlled conditions, the formation of intermetallic iron-containing compounds in aluminium alloy melts, and thereby determine the optimized conditions for carrying out the refining of melts of aluminium scrap material containing varying amounts of iron, and having varying target levels of iron after refining. From these test examples there was derived the insight that the method of the invention can be operated successfully to achieve the desired result in terms of low Mn usage and precise Fe reduction. It furthermore became apparent that a particular Si level, e.g. 8% or 12% can be maintained.

#### Example 1

A melt of 12 kg was composed in an induction furnace. The melt consisted of (in percent by weight): 12.1% Si, 0.83% Fe, 0.32% Cr, 0.41% Ti, 0.23% Zr, 0.01% Mo, balance aluminium (and other inevitable impurities). The different elements were supplied via AlSi20, AlFe50, AlZr10, AlCr20, FeMo80 master-alloys and technically pure aluminium (A199.7). The melt was heated to 855°C and held at that temperature for 30 minutes to allow all of the master-alloys to dissolve. Subsequently the melt was cooled to 605°C and held at this temperature for 20 minutes. During cooling and the holding time, intermetallic compounds are formed and will partly deposit into the melt. Afterwards the melt was poured into a preheated filter box with a ceramic foam filter (CFF) with a filter porosity of 20 ppi. The filtrate was analyzed

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and consisted of: 11.9% Si, 0.62% Fe, 0.15% Cr, 0.12% Ti, 0.09% Zr, traces of Mo, balance aluminium.

### Example 2

In a manner identical to that of example 1, a melt was made from a composition of (in percent by weight) : 11.5% Si, 0.78% Fe, 0.37% Mn, 0.32% Cr, 0.40% Ti, 0.26% Zr, 0.01% Mo, balance aluminium. Using the same process parameters (30 minutes homogenizing at 855°C, cooling to 605°C, holding time 20 minutes, poured onto a 20 ppi CFF filter), following filtration the melt consisted of: 11.4% Si, 0.49% Fe, 0.19% Mn, 0.11% Cr, 0.11% Ti, 0.10% Zr, traces of Mo, balance aluminium.

### Example 3

In a manner identical to that in examples 1 and 2, a melt was made from a composition of (in percent by weight): 12.6% Si, 0.87% Fe, 0.21% Cr, 0.11% Ti, 0.14% Zr, balance aluminium. Using the process parameters (30 minutes homogenizing at 850°C, cooling to 630°C, holding time 20 minutes, poured onto a 30 ppi CFF filter), following filtration the melt consisted of: 12.8% Si, 0.85% Fe, 0.20% Cr, 0.11% Ti, 0.14% Zr, balance aluminium.

### Examples 4-22

In a manner identical to that in example 1, a number of melts were made whose initial compositions are given in Table 1. The process conditions used are given in Table 2. In all cases the melt was first homogenized for 30 minutes at temperature between 850-860°C. The composition obtained in examples 4-22 following refining is given in Table 3.

Tables 1-3 below give respectively the initial composition, the process parameters used and the final composition of the melt for examples 4-22, all amounts being % by weight.

### Example 23

For the alloy with the initial composition: 12% Si, 2% Fe, 1.5% Mn, 0.2% Cr, balance aluminium, the iron removal yield is determined as a function of the process parameters:

- holding temperature
- holding time
- filter porosity.

The results are expressed graphically, and discussed below.

Table 1

Initial composition for examples 4-22 (balance aluminium)			
Example	Si	Fe	Mn
4	11.5	1.12	0.99
5	11.4	1.09	1.86
6	11.4	1.62	0.99
7	11.2	1.58	1.90
8	11.3	2.09	0.99
9	11.6	2.07	1.91
10	12.0	1.89	1.57
11	< 0.1	1.09	0.49
12	< 0.1	1.09	0.98
13	< 0.1	1.08	1.45
14	< 0.1	1.58	0.49
15	< 0.1	1.48	0.96
16	< 0.1	1.52	1.45
17	< 0.1	1.56	1.44
18	< 0.1	1.91	0.95
19	8.19	1.90	0.58

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Table 1 (continued)

Initial composition for examples 4-22 (balance aluminium)			
Example	Si	Fe	Mn
20	8.20	1.50	0.85
21	8.27	1.22	1.12
22	8.25	0.57	1.57

Table 2

Process parameters used for examples 4-22			
Example	Holding time	Holding temperature	Filter type
4	15 min	605°C	10 ppi
5	15 min	605°C	10 ppi
6	15 min	605°C	10 ppi
7	15 min	605°C	10 ppi
8	15 min	605°C	10 ppi
9	15 min	605°C	10 ppi
10	15 min	605°C	10 ppi
11	15 min	665°C	10 ppi
12	15 min	665°C	10 ppi
13	15 min	665°C	10 ppi
14	15 min	665°C	10 ppi
15	15 min	665°C	10 ppi
16	15 min	665°C	10 ppi
17	15 min	680°C	10 ppi
18	15 min	685°C	10 ppi
19	30 min	630°C	30 ppi
20	30 min	630°C	30 ppi
21	30 min	640°C	30 ppi
22	30 min	640°C	30 ppi

Table 3

Final composition for examples 4-22 (balance aluminium)			
Example	Si	Fe	Mn
4	11.7	0.64	0.42
5	11.6	0.47	0.58
6	11.7	0.82	0.35
7	11.3	0.61	0.50
8	11.5	1.02	0.31
9	12.2	0.85	0.53
10	12.1	0.76	0.40
11	< 0.1	1.10	0.49
12	< 0.1	1.10	0.98
13	< 0.1	0.87	1.15
14	< 0.1	1.59	0.49
15	< 0.1	1.25	0.77
16	< 0.1	1.14	1.03
17	< 0.1	1.12	0.99
18	< 0.1	1.57	0.74

Table 3 (continued)

Final composition for examples 4-22 (balance aluminium)			
Example	Si	Fe	Mn
19	8.24	1.30	0.31
20	8.50	1.00	0.44
21	8.44	0.79	0.63
22	8.51	0.33	0.80

## BRIEF INTRODUCTION OF THE DRAWINGS

The results of the tests in accordance with the examples are illustrated graphically in the accompanying figures:-

Fig. 1 shows the initial and final concentrations of an Al-Si<sub>12</sub>-Fe-Mn system,  
 Fig. 2 shows the final concentrations of an Al-Si<sub>12</sub>-Fe-Mn system,  
 Fig. 3 shows the initial and final concentrations of an Al-Fe-Mn system,  
 Fig. 4 shows the final concentrations of an Al-Fe-Mn system,  
 Fig. 5 shows the initial and final concentrations of an Al-Si<sub>8</sub>-Fe-Mn system,  
 Fig. 6 shows the final concentrations of an Al-Si<sub>8</sub>-Fe-Mn system,  
 Fig. 7 sets out the Mn/Fe ratio in the refined melt plotted against the slope of the lines in Figs. 1, 3 and 5,  
 Fig. 8 sets out the Fe removal ratio plotted against the filter type at a holding temperature of 605°C for 30 minutes,  
 Fig. 9 sets out the Fe removal ratio plotted against the holding time, i.e. the time as shown in Table 2, at a holding temperature of 605°C, and  
 Fig. 10 sets out the metal yield, i.e. the weight of the refined melt following filtration relative to the weight of the melt to be refined plotted against the holding temperature, i.e. the temperature as shown in Table 2.

## DESCRIPTION OF THE FIGURES

Figs. 1 and 2 give for examples 4-10 the initial and final compositions respectively for the Fe and Mn content. The initial and final points of each example are linked together by a straight line in Fig. 1.

Figs. 3 and 4 give for examples 11-18 the initial and final compositions respectively for the Fe and Mn content. Here too the points are linked together for each example by straight lines, in Fig. 3.

Figs. 5 and 6 give for examples 19-22 the initial and final compositions respectively for the Fe and Mn content. The respective points for each example are linked together by a straight line in Fig. 5.

The final compositions in Fig. 1 lie within a certain margin along a straight line when plotted in Fig. 2. This also applies for the final compositions in Figs. 3 and 5, as plotted in Figs. 4 and 6.

Fig. 7 illustrates the slope of the straight lines from Figs. 1, 3 and 5 as a function of the initial ratio Mn/Fe. Therefore the slope is a function of the ratio Mn/Fe and the Si content. From this there is derived the insight that the final Fe content can be accurately obtained by adjustment of initial Mn content.

The results of yield measurements in example 23 are illustrated graphically in Fig. 8 (as a function of the filter porosity), Fig. 9 (as a function of the holding time), and Fig. 10 (as a function of the holding temperature).

The Fe removal yield here is the Fe removal ratio (final Fe level relative to initial Fe level). The Fe removal yield ( $\eta_{Fe}$ ) as a function of the filter porosity in ppi may be expressed as  $\eta_{Fe} = 57.4 + 0.21 p$ , where p is the filter porosity in ppi at a holding temperature T of 605°C for t = 30 minutes. Furthermore the following relations are found to exist:

$$\eta_{Fe} = 38.8 + 11.07 t - 1.31 t^2 \text{ where } t = \text{holding time in min at a holding temperature of } 605^\circ\text{C};$$

$$\eta_{Fe} = 60.96 - 0.2 T \text{ where } T = \text{holding temperature in } ^\circ\text{C, at a holding time of 30 min. and a } p = 30 \text{ ppi.}$$

Thus each process may be optimized according to these parameters.

## Claims

1. Method of refining a melt of aluminium scrap material which comprises metallic aluminium and impurities including iron, comprising the steps of

- (i) determining the initial amounts, in % by weight, in the composition of said melt of aluminium scrap material of the elements Mn, Fe and Si, these amounts being identified below as  $[Mn_0]$ ,  $[Fe_0]$  and  $[Si_0]$ ;  
(ii) adding a quantity  $Mn_x$  of Mn to the melt, so as to obtain in the melt, after refining of the melt by steps (iii), (iv) and (v) below, a ratio  $\delta$  given by

$$\delta = [Mn_1] / [Fe_1]$$

wherein  $[Mn_1]$  and  $[Fe_1]$  are the amounts in % by weight of Mn and Fe in the melt after the refining of the melt,  $[Fe_1]$  being a desired target level of Fe and  $[Mn_1]$  being given by

$$[Mn_1] = A - B * [Fe_1]$$

wherein

$$1.86 - 0.17 * [Si_0] + 0.004 * [Si_0]^2 < A < 2.21 - 0.17 * [Si_0] + 0.005 * [Si_0]^2$$

and

$$0.42 + 0.50 * \exp(-0.28 * [Si_0]) < B < 0.57 + 0.50 * \exp(-0.28 * [Si_0]),$$

said added quantity  $Mn_x$  of Mn being given by

$$Mn_x = \delta * Fe_0 - Mn_0$$

- wherein  $Fe_0$  and  $Mn_0$  are the initial quantities of Fe and Mn in said melt of aluminium scrap material;  
(iii) homogenizing the melt by heating after step (ii) ;  
(iv) cooling the melt after step (iii) and maintaining the melt at a super-eutectic holding temperature T for a holding time t so that solid intermetallic compounds form;  
(v) separating the solid intermetallic compounds from the melt, to obtain the refined melt.

2. A method in accordance with claim 1, wherein

$$0.45 + 0.50 * \exp(-0.28 * [Si_0]) < B < 0.50 + 0.50 * \exp(-0.28 * [Si_0]).$$

3. A method in accordance with claim 1 or 2, wherein the melt is of the Al-Si12-Fe-Mn type and A lies between 0.76 and 0.80 and B is approximately 0.49.
4. A method in accordance with claim 1 or 2, wherein the melt is of the Al-Fe-Mn type and A lies between 2.00 and 2.04 and B is approximately 0.96.
5. A method in accordance with claim 1 or 2, wherein the melt is of the Al-Si8-Fe-Mn type and A lies between 0.97 and 1.01 and B is approximately 0.52.
6. A method in accordance with any one of claims 1 to 5, wherein the separating step (v) is performed in a filter having a filter porosity p less than 30 ppi (pores per square inch).
7. Aluminium alloy obtained from a refined melt produced by the method of any one of claims 1 to 6.



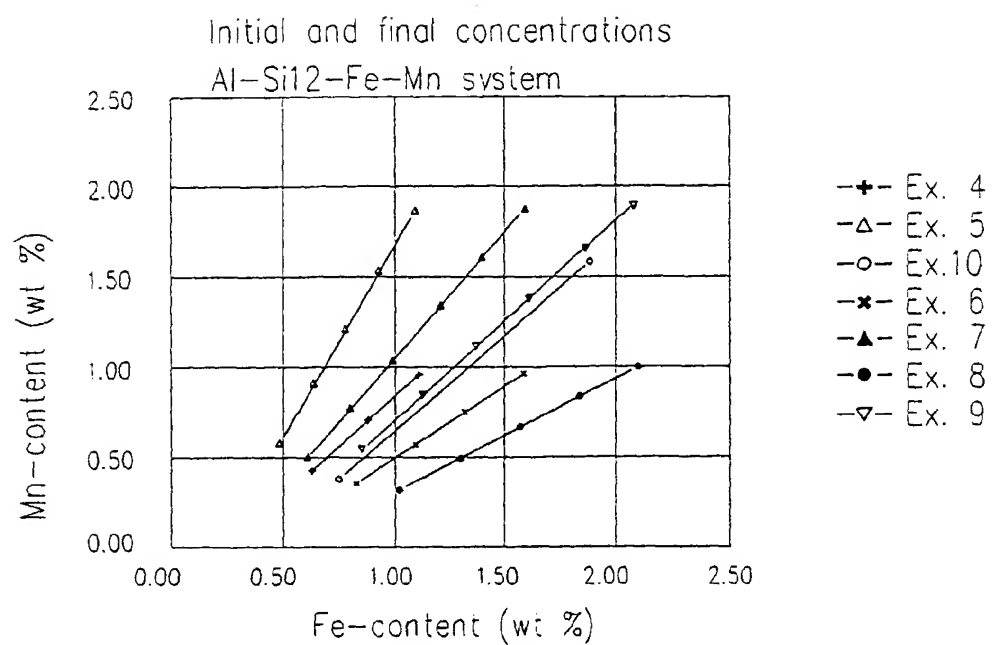


Fig. 1

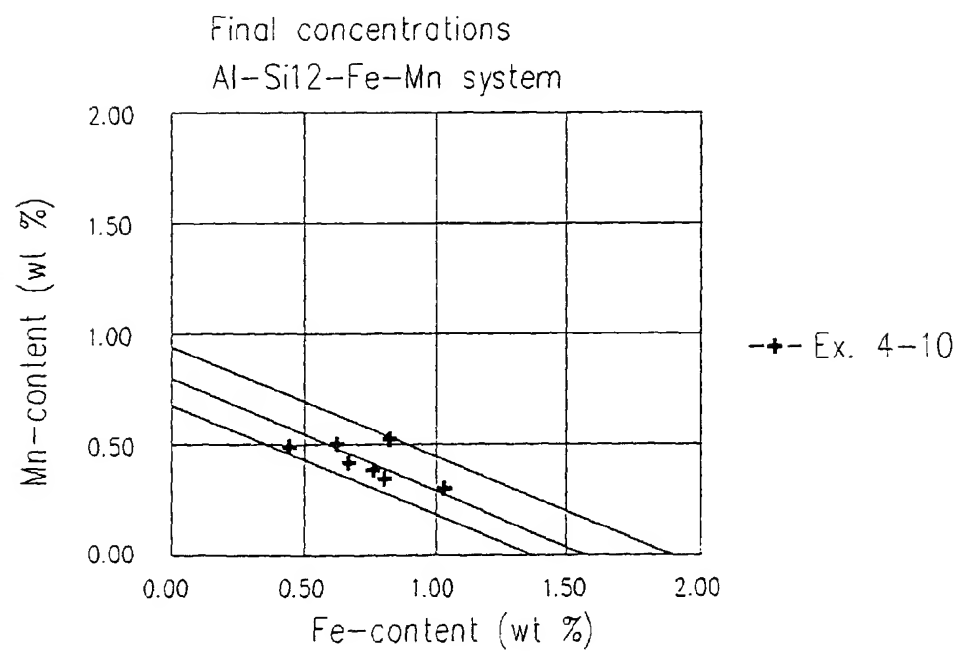


Fig. 2

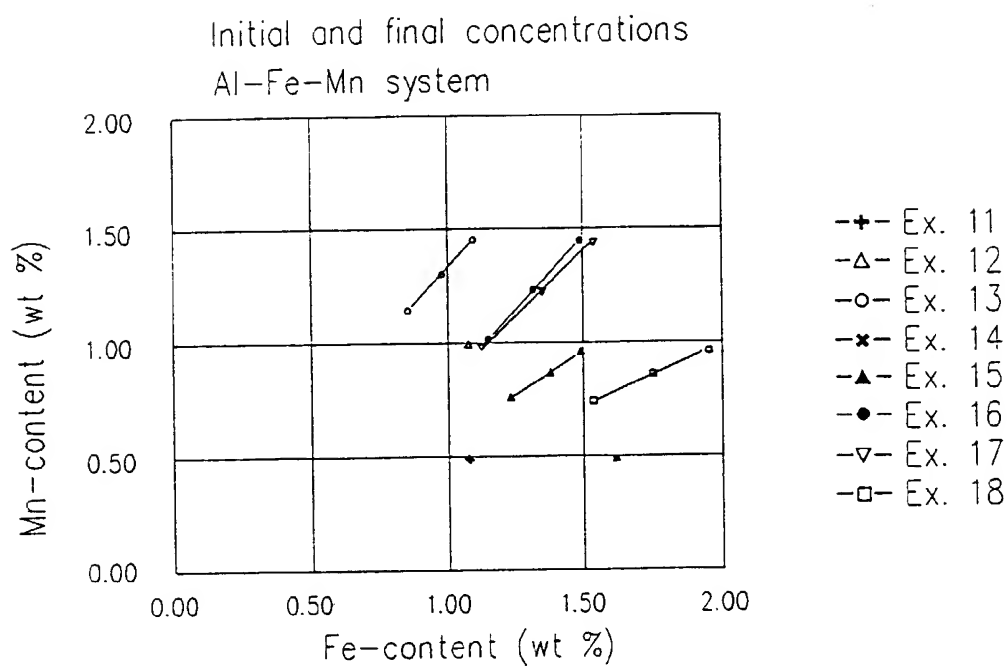


Fig. 3

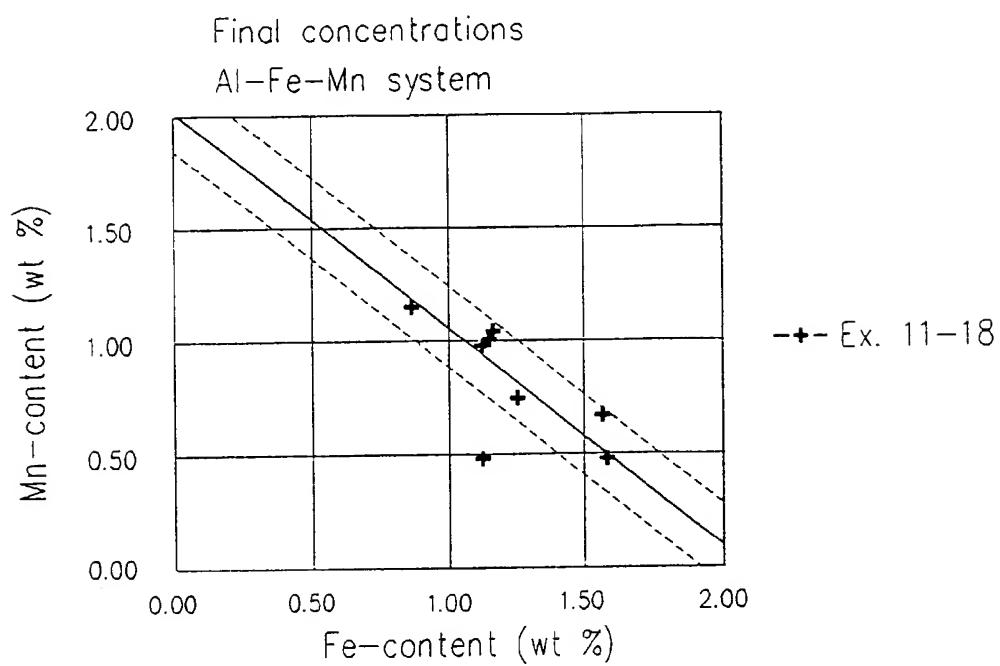


Fig. 4

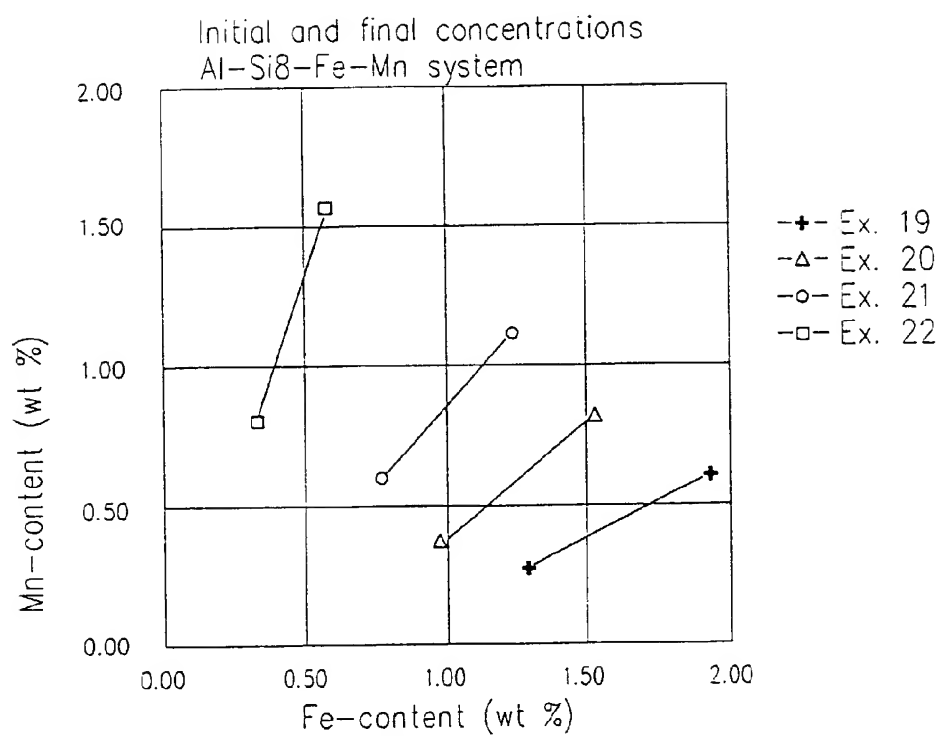


Fig. 5

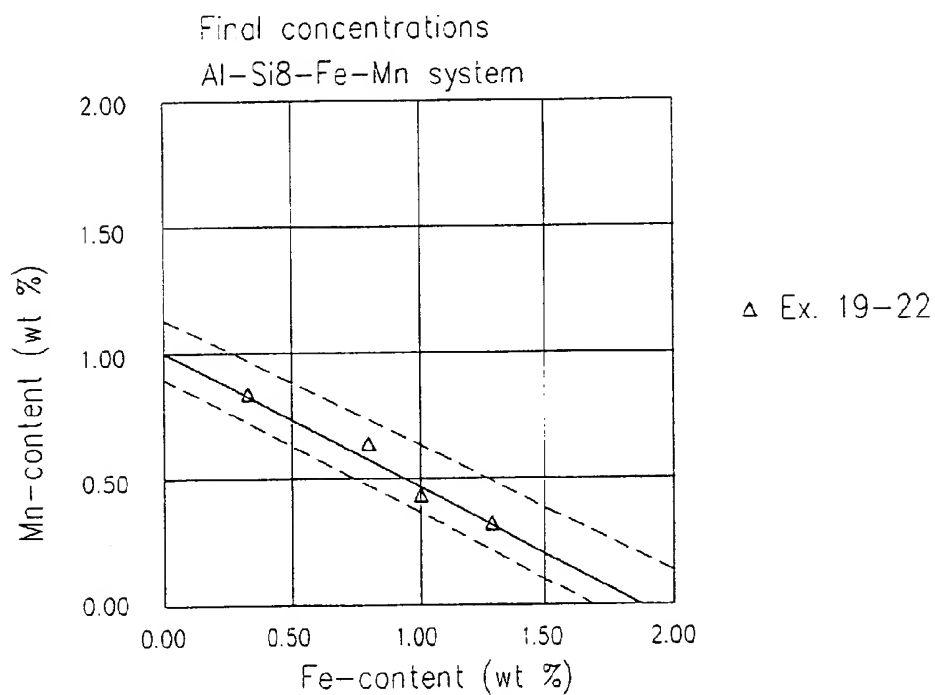


Fig. 6

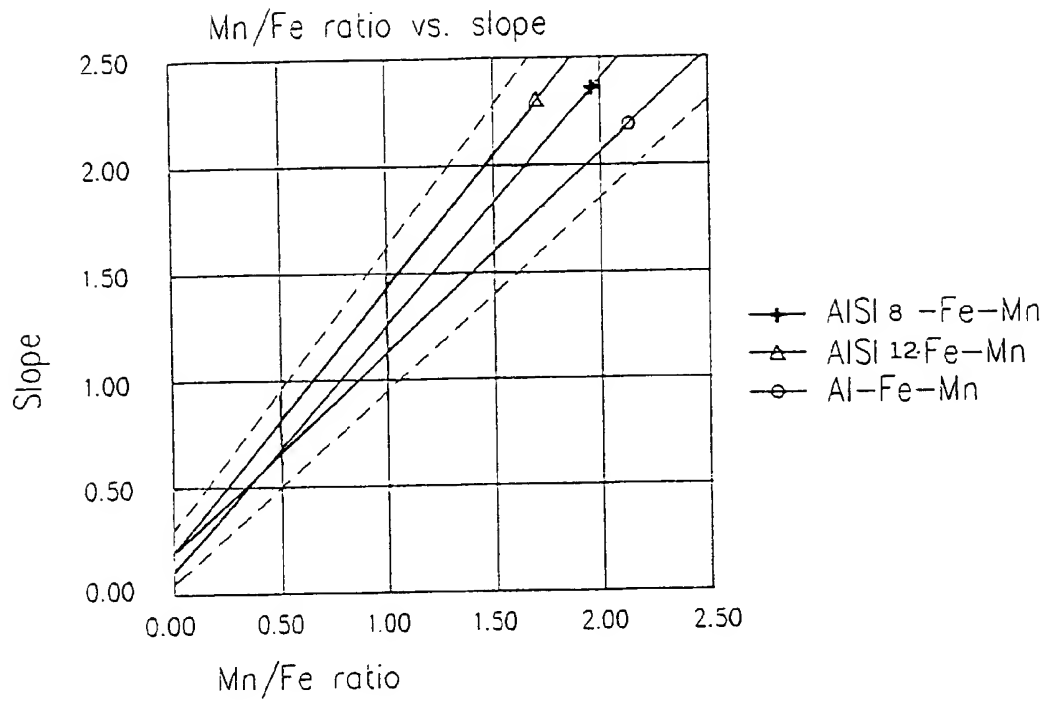


Fig. 7

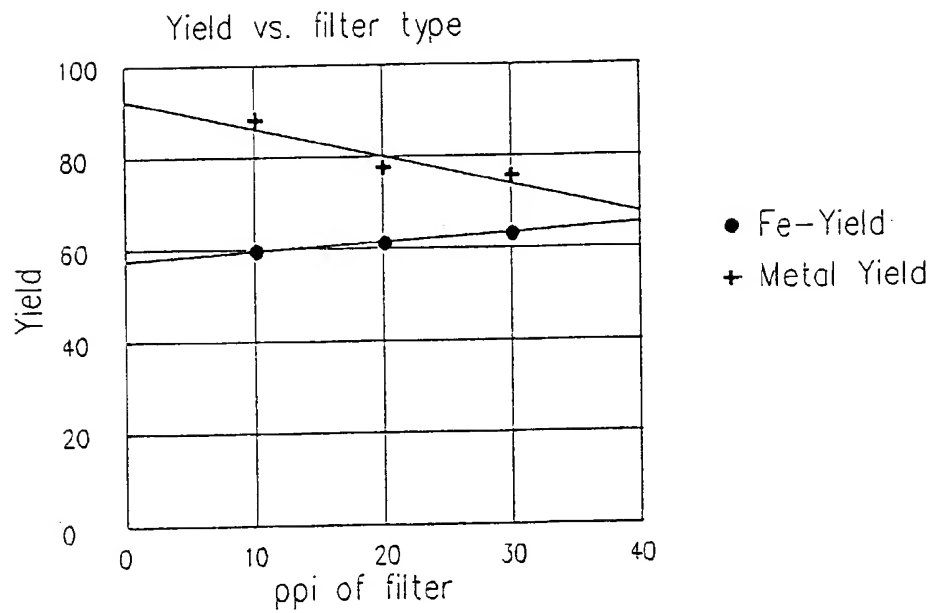


Fig. 8

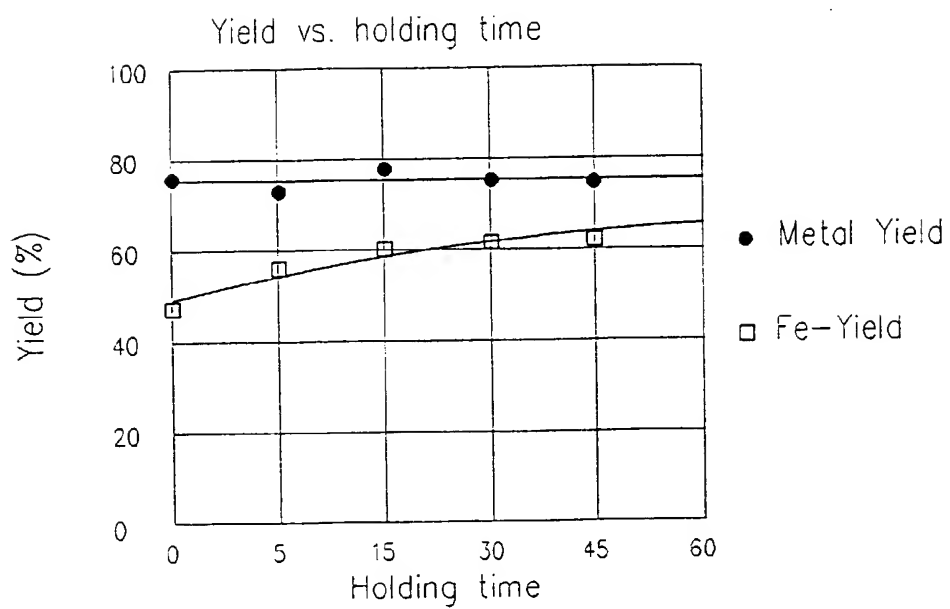


Fig. 9

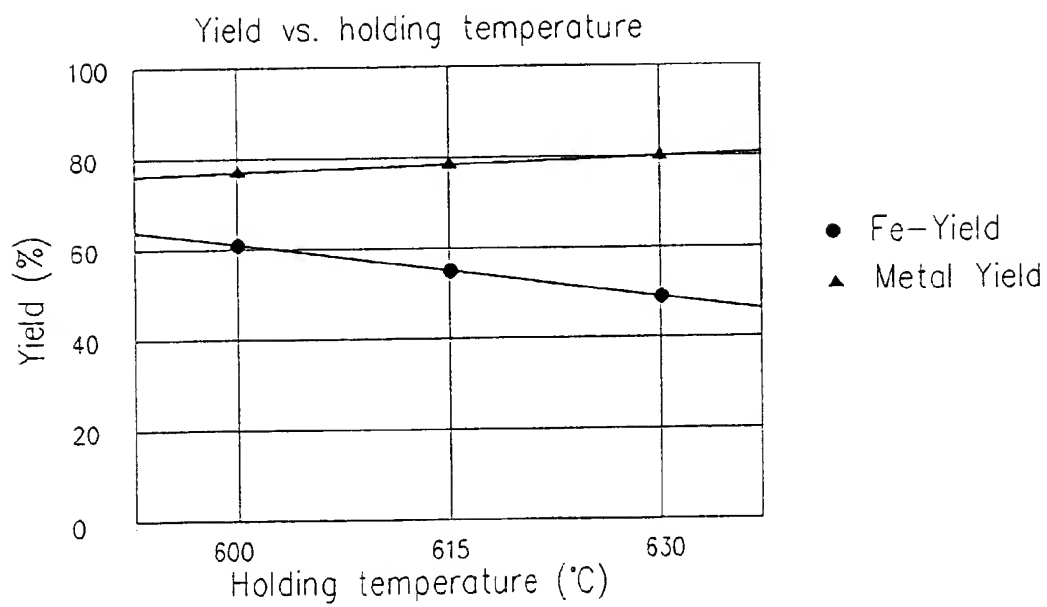


Fig. 10



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## EUROPEAN SEARCH REPORT

Application Number  
EP 96 20 1498

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Category	Citation of document with indication, where appropriate, of relevant passages	Relevant to claim	CLASSIFICATION OF THE APPLICATION (Int.Cl.6)
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A,D	DATABASE WPI Section Ch, Week 8510 Derwent Publications Ltd., London, GB; Class M26, AN 85-061392 XP002012261 & SU-A-1 108 122 ( DNEPR METAL INST) , 15 August 1984 * abstract *	1,6	
A,D	PATENT ABSTRACTS OF JAPAN vol. 010, no. 103 (C-340), 18 April 1986 & JP-A-60 234930 (TOYOTA JIDOSHA KK), 21 November 1985, * abstract *	1,3	
A,D	DATABASE WPI Section Ch, Week 9519 Derwent Publications Ltd., London, GB; Class M25, AN 95-145191 XP002012262 & JP-A-07 070 666 ( NIKKEI GIKEN KK/NIPPON LIGHT METAL CO) , 14 March 1995 * abstract *	1,3	
			TECHNICAL FIELDS SEARCHED (Int.Cl.6)
			C22B
The present search report has been drawn up for all claims			
Place of search THE HAGUE		Date of completion of the search 2 September 1996	Examiner Wittblad, U
CATEGORY OF CITED DOCUMENTS		T : theory or principle underlying the invention E : earlier patent document, but published on, or after the filing date D : document cited in the application L : document cited for other reasons & : member of the same patent family, corresponding document	
X : particularly relevant if taken alone Y : particularly relevant if combined with another document of the same category A : technological background O : non-written disclosure P : intermediate document			

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A,D	--- PATENT ABSTRACTS OF JAPAN vol. 940, no. 010 & JP-A-06 299265 (NIPPON LIGHT METAL CO LTD;OTHERS: 01), 25 October 1994, * abstract *	1	
A,D	--- PATENT ABSTRACTS OF JAPAN vol. 950, no. 002 & JP-A-07 054070 (NIPPON LIGHT METAL CO LTD), 28 February 1995, * abstract *	1	
T	--- Z. METALLKUNDE, vol. 86, no. 7, MÜNCHEN, DE, pages 457-464, XP002012260 LAKSHMANAN ET AL: "Microstructure Control of Iron Intermetallics in Al-Si Casting Alloys" * page 457 - page 464 * -----	1-5	
The present search report has been drawn up for all claims			TECHNICAL FIELDS SEARCHED (Int.Cl.6)
Place of search THE HAGUE		Date of completion of the search 2 September 1996	Examiner Wittblad, U
<p><b>CATEGORY OF CITED DOCUMENTS</b></p> <p>X : particularly relevant if taken alone Y : particularly relevant if combined with another document of the same category A : technological background O : non-written disclosure P : intermediate document</p> <p>T : theory or principle underlying the invention E : earlier patent document, but published on, or after the filing date D : document cited in the application L : document cited for other reasons ----- &amp; : member of the same patent family, corresponding document</p>			

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